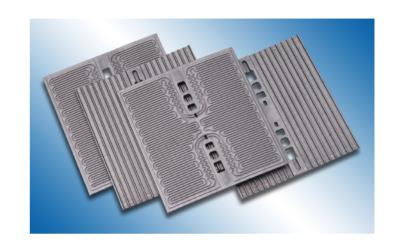
# 2008 DOE Hydrogen Program Review Next Generation Bipolar Plates for Automotive PEM Fuel Cells



Orest Adrianowycz, Ph.D. GrafTech International Ltd. June 11<sup>th</sup>, 2008

**Project ID # FC28** 











#### **Overview**

#### **Project Timeline**

- Start date: March 1st, 2007
- End date: February 29th, 2009
- Percent complete: 50%

#### **Budget**

- Total project: \$2.9 MM
  - DOE share: \$2.3 MM
  - Contractor share: \$0.6MM
- Spending FY07: \$760K
- Budgeted FY08: \$1,550K

#### **Barriers** (bipolar plates)

- A Durability
  - Improved corrosion resistance
  - Decrease weight and volume
- B Cost
  - Lower material & production costs
  - Increased power density
- C Performance
  - Improved gas impermeability
  - Improved electrical and thermal conductivity



#### **Collaborators**

# **GrafTech International Ltd. Primary Contractor**

- Orest L. Adrianowycz, Ph.D. Principle Investigator
- Expanded graphite selection
- Preparation and testing of intermediate polymer-graphite composites
- Final graphite/polymer composition selection
- Bipolar plate manufacturing

#### **Ballard Power Systems**

- Warren Williams, Team Leader
- Flow field plate design
- Fuel cell stack assembly
- Durability and freeze-start testing
- Post-test analysis of composite plates

#### **Huntsman Advanced Materials**

- Roger Tietze, Team Leader
- Preliminary screening of high temperature resin formulations
- Mold release agents and flow additives for the resin formulations
- Scale-up of resin production for full-size plates manufacturing

#### **Case Western Reserve University**

- Professor Tom Zawodzinski, Team Leader
- High temperature membrane materials and testing protocol selection
- High temperature single cell testing of resin-graphite composite flow field plates
- Post-test analysis of high temperature single cell effluent



## **Program Objectives**

#### **Overall Project Objective**

 Develop next-generation automotive bipolar plates based on an engineered composite of expanded graphite and resin capable of operation at 120 °C

#### **Goals Year 1**

- Develop graphite/polymer composite to meet 120°C fuel cell operating temperature
- Demonstrate manufacturing capability of new materials to a reduced bipolar plate thickness of 1.6 mm

#### Goals Year 2

- Manufacture high-temperature flow field plates for full scale testing
- Validate performance of new plates under automotive conditions using a short (10-cell) stack
- Show viability of published cost target through the use of lowcost materials amenable to high volume manufacturing



## **Program Milestones/Go-No Go Decisions**

Task	Milestones	Target Date	Status
1	Expanded Graphite Material Selection		
1.2.7	Final Graphite Flake Sources Selected	03/23/07	Complete
1.3.2.9	Graphite for resin evaluation Identified		Complete
1.3.3.9	Experimental Graphite Resin Evaluation Completed	01/02/08	In progress
2	Resin Identification and Selection		
2.7	Resin Formulations for Composite Studies Selected	05/21/07	Complete
3	Small-Scale Composite Preparation and Evaluation		
3.2.10	Resins for Single Cell Testing Selected	08/30/07	Complete
3.2.12	Contingency Point: Resins for Single Cell Testing Selected	08/30/07	Complete
4	Machining and Embossment of Small-Scale Composites		
4.3.5	Machined Plates Completed	10/12/07	Complete
4.5.2	Composites Embossability Characterized	01/16/08	Complete
4.6.5	Final Graphite, Resin and Processing Parameters Selected	03/17/08	Complete
5	Single Cell Testing		
5.10	Single Cell Testing Completed	03/13/08	In progress
6	Design and Manufacture Full-size Bipolar Plates		
6.5.7	Full Size Tool and Leak Check Device Ready	09/03/08	In progress
6.6.18	Full Size Plates Ready for Short Stack Testing	10/01/08	Not Started
7	Short Stack Test of Full-size Plates		
7.1.5	Short Stack Full Size Plates Ready for Testing	11/05/08	Not Started
7.3.3	Final Review of Short Stack Test Results Completed	02/20/09	Not Started
7.4.2	Stack Delivered to DOE	03/06/09	Not Started
8	Economic Assessment of New Technologies		
8.2	Economic Assessment Complete	12/16/08	Not Started



## **Approach**

#### **Task 1: Graphite Selection**

- Raw Material Evaluation
- Intercalation Chemistry and Processing Optimization

#### **Task 2: Resin Selection**

- Resin Evaluation
- Resin Selection

#### Task 3: Small-Scale Composite Prep

- Develop Methods for Composite Plate Manufacturing and Testing
- Evaluate Thermal and Mechanical Properties
- Conduct Environmental Chamber Testing

# Task 4: Composite Machining and Embossing

- Machined Plates for Single Cell Testing
- Validate Composite Properties
- Evaluate Small Embossed Test Plates

#### **Task 5: Single Cell Testing**

- Select High Temp Cell Components
- Develop Test Method for Leachates
- Perform Single Cell Testing and Analysis

## Task 6: Design and Manufacture Full-size Bipolar Plates

- Design Flow Field Plate Molds
- Fabricate Full Size Embossing Die Set
- Manufacture Full-size Bipolar Plates

#### Task 7: Full-size Plate Short Stack Testing

- Short stack plate assembly
- Test Cells in Short-Cell Stack
- Post-Test Analysis
- Deliver Full Size Plate Stack to DOE

## Task 8: Economic Assessment of New Technologies

 Perform economic assessment of the selected raw material and manufacturing processing



#### Flow Field Plate Functions

- Serves as a current collector
- Distributes fuel (typically hydrogen) and oxidant (typically air) uniformly over the cell active areas
- Facilitates water management of the membrane to keep it humidified
- Acts as an impermeable barrier to maintain the hydrogen gradient across the membrane necessary for high power output
- Provides some structural support for the stack
- Removes heat from the active area of the cells
- The most bulky component in the PEM fuel cell stack, by both weight and volume



#### **DoE Targets for Bipolar Plate Performance**

Characteristic	Units	Status 2005 <sup>a</sup>	2010 Target	2015 Target
Cost <sup>b</sup>	\$/kW	10 <sup>c</sup>	5	3
Weight	kg/kW	0.36	<0.4	<0.4
H <sub>2</sub> Permeation Flux	cm <sup>3</sup> sec <sup>-1</sup> cm <sup>-2</sup>	< 2 x 10 <sup>-6</sup>	< 2 x 10 <sup>-6</sup>	< 2 x 10 <sup>-6</sup>
@ 80 °C, 3 atm. (equivalent to <0.1 mA/cm <sup>2</sup> )				
Corrosion	μA/cm <sup>2</sup>	<1 <sup>d</sup>	<1 <sup>d</sup>	<1 <sup>d</sup>
Electrical Conductivity	S/cm	>600	>100	>100
Resistivity <sup>e</sup>	Ohm cm <sup>2</sup>	<0.02	0.01	0.01
Flexural Strength <sup>f</sup>	MPa	>34	>25	>25
Flexibility	% deflection at mid-span	1.5 to 3.5	3 to 5	3 to 5

#### (DoE Publication Table 3.4.14)

- This is the first year for which status is available. 2005 status is for carbon plates, except for corrosion status which is based on metal plates.
- b Based on 2002 dollars and costs projected to high volume production (500,000 stacks per year).
- Status is from 2005 TIAX study and will be periodically updated.
- d May have to be as low as 1 nA/cm if all corrosion product ions remain in ionomer.
- Includes contact resistance.
- f Developers have used ASTM C-651-91 Standard Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Four Point Loading at Room Temperature.



# Flow Field Plate Technologies Comparison

Technology	Advantages	Disadvantages
<b>GRAFCELL®</b> Resin	Chemically inert	Strength
Impregnated	Electrical conductivity	Not as thin as metals
Flexible Graphite	Contact resistance	
	Thermal conductivity	
	Thin	
	Lower Cost	
	Proven performance	
<b>Graphite-Filled</b>	Known fabrication techniques	Thermal conductivity
Polymers	Molded-in flow fields	Electrical conductivity
	Low Density	Temperature capability
		Brittleness
		Molding with high filler content
Metals	Electrical conductivity	Corrosion
	Strength Temperature	Poisoning of MEA
	Thin	Contact resistance
	Known fabrication techniques	Thermal conductivity
		Density
		<b>Expensive alloys and coatings</b>



## **Property Status for New Expanded Graphite FFPs**

<b>Material Property</b>	DOE Target Need	Status
Electrical Conductivity	Plate	Meets DoE 2015 Target
Thermal Conductivity	Balance of Plant	Meets DoE 2015 Target
Contact Resistance	Plate	Meets DoE 2015 Target
Temperature (120 °C)	MEA, Balance of Plant	R&D needed
Gas Impermeability	Plate	R&D needed
Mechanical Strength	Plate	R&D needed
Corrosion	Plate	Meets DoE 2015 Target
Ion Leachability	System Durability	R&D needed
Thickness	System Power Density	R&D needed
Manufacturability	Technology Viability	R&D needed
Flexibility	Plate	Meets DoE 2015 Target
Weight	Plate	Meets DoE 2015 Target
Cost	All	R&D needed



#### **Approach to Meeting Remaining DOE FFP Targets**

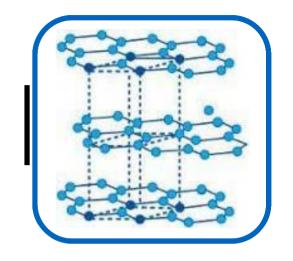
#### The "4 T's":

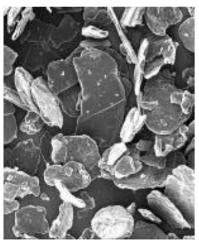
- Temperature
  - New higher temperature resin systems
- Thickness
  - Enabled through a combination of flake sourcing, flake processing, and composite processing
- Toughness
  - Enhanced through resin-reinforcement
- Throughput
  - Viable path to commercialization brought about by low cost manufacturing methodologies

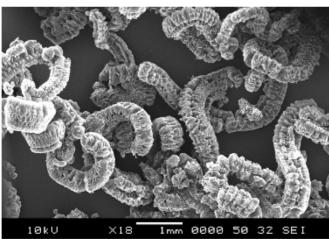


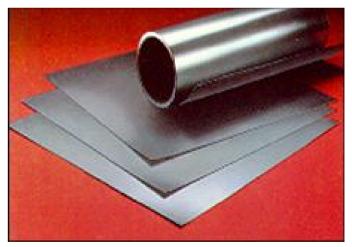
# **Expanded Graphite**

- Graphite flake is expanded by chemically "inserting" certain compounds between the graphite planes (intercalation)
- When exposed to heat, the chemicals inside the graphite decompose, forcing the graphite layers apart (exfoliation)





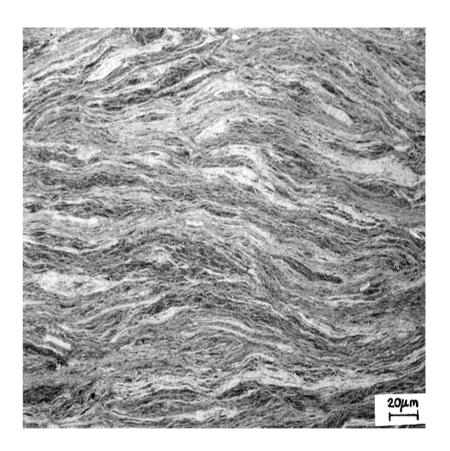






## **Expanded Graphite Sheet**

- Calendaring, embossing, pressing
  - No binder required to make a continuous sheet
- Compression of expanded graphite re-aligns graphite layers
  - Structurally anisotropic
- Impregnable, Conformable, Sealable
- Light weight
- Thermally and Electrically Conductive – continuous graphite phase
- Near-zero CTE (Coefficient of Thermal Expansion) in-plane
- Chemically inert



Layers in compressed graphite sheet



## **Accomplishments – Task 1**

#### **Expanded Graphite Material Selection**

Contributors: GrafTech, Ballard, Huntsman

Subtask 1.1: Define key flow field plate specifications with collaborators (Complete)

**Subtask 1.2: Natural Graphite Selection (Complete)** 

 Natural graphite sources from a number of domestic and international suppliers were evaluated. Candidate flakes from these sources were selected.

Subtask 1.3: Intercalation Chemistry and Exfoliation Methods (In Progress)

- Design of experimental methodology
- Initial screening experiment complete. Preliminary materials selected for further study.
- Response surface experiment to identify interactions and final usable range of materials in progress.



# Accomplishments – Task 2 (Complete)

#### Resin Identification and Selection

Contributors: GrafTech, Huntsman

**Subtask 2.1: Resin Specifications Defined** 

- Based on key fuel cell performance characteristics
- Table of resin specifications developed

**Subtask 2.2: Potential New Resin Chemistries Definition** 

- High performance epoxy and benzoxazine resins selected
- Subtask 2.3: Part Release Chemistry Design
- Mold release chemistry incorporated into resin formulations
- Subtask 2.4: Lab Scale Resin Samples Formulated and Neat Resin Properties Evaluated
- 9 Benzoxazine and 6 Epoxy formulations evaluated
- **Subtask 2.5: Resin Formulations Down select (Milestone)**
- 2 Benzoxazine and 1 Epoxy resins down selected



#### **Accomplishments - Task 2: Neat Resin Systems**

				DMA	A Tg	TN	ΛA	TO	SA .
		Gel Time	Softening		Storage			Decomp	Weight
System	Catalyst	@ 200°C	Point	Tan Delta	Modulus	Tg	CTE	Temp	Loss
		S	°C	°C	°C	°C	μm/m°C	°C	%
Benzoxa	Benzoxazine Resin								
1	No	>600	70.5	215	185	183	64	339	2.2
2	No	>600	84.8	171	137	128	82	319	1.9
2A	No	>600	Liquid	232	198	216	85	351	3.1
2B	No	>600	Liquid	225	183	195	159	343	2.6
2G	No	364.9	88.1	282	252	247	61	343	3.8
2H	No	440.9	74.6	282	255	261	52	347	3.8
3	No	>600	80.5	298	183	175	67	317	2.5
4	No	420	98	148	120	114	75	NA	NA
5	No	>600	87	183	148	104	65	NA	NA
<b>Epoxy Re</b>	esin								
1	Yes	30.3	Liquid	205.0	178.0	172.0	82.0	336.0	3.8
2	Yes	170/150°C	Liquid	208.0	191.0	184.0	81.0	309.0	3.5
3	Yes	100/150°C	Liquid	242.0	210.0	197.0	72.0	341.0	3.5
4	No	155/150°C	Liquid	156.0	125.0	NA	NA	NA	NA
5	Yes	31.3	Liquid	143.0	96.0	NA	NA	286.0	3.2
6	No	>600	Liquid	95.0	88.0	NA	NA	NA	NA
	Selected	Systems							



# Accomplishments – Task 3 (Complete)

**Small-Scale Composite Preparation** 

**Contributors: GrafTech, Huntsman** 

Subtask 3.1: Prepare Flexible Graphite Mat for Resin Evaluation Subtask 3.2: Preliminary Composite Preparation and Evaluation

- Un-embossed expanded graphite-resin composites successfully fabricated
- Composite molding temperature defined
- Gas impermeability verified
- Resins selected for single cell and embossing studies (Milestone)
  - Epoxy resin system eliminated due to processing issues

**Subtask 3.3: Long-Term Testing of Selected Composite Samples** 

- Flexural and tensile strength and modulus are improved or not significantly changed during both cycling and shock exposure for 2G resin system.
- The 2H system 3-ply samples show some degradation in strength Subtask 3.4: Composite Processing Variable Experimental Design Conducted
- Key process variables identified
- Optimization study conducted on embossed plates



#### **DMA Analysis of 2G Resin Graphite Composite**

Sample: 664-15-39-1 2G1Ply-2 2008-02970 Size: 17.5000 x 12.7900 x 0.6200 mm

Method: Ramp 25 to 280C Cycled

Comment: 0.2mmOA, 3inlb, 2C m Cycled 2008040904

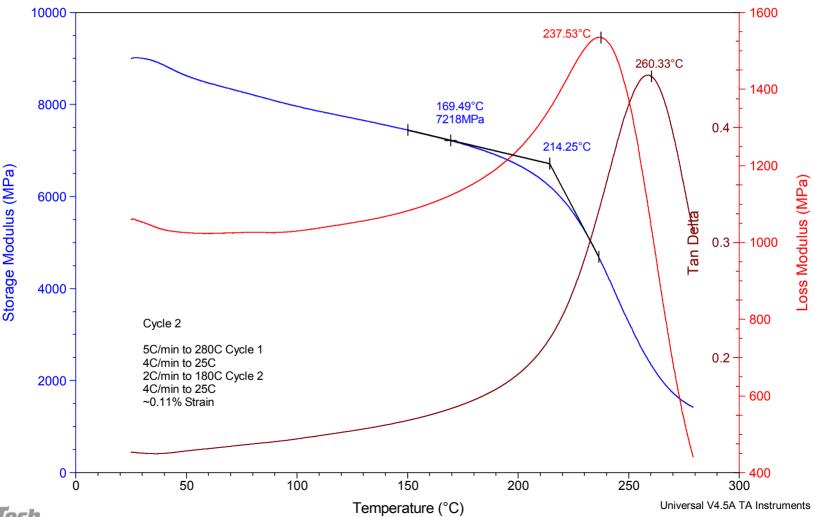
**DMA** 

File: 664-15-39-1 2G1Ply-2 2008-02970.001.001

Operator: D.M. Riffle

Run Date: 15-Apr-2008 09:37

Instrument: DMA Q800 V7.5 Build 127





## **Subtask 3.2 Composite Property Comparison**

			FFP	2G Resin	2H Resin
Property	Method	Units	Average	Average	Average
<b>Bulk Density</b>	ASTM C611	g/cm3	1.68	1.68	1.72
Thermal Conductivity (x,y)	ASTM D5470 Modified	W/m-K	275	286	294
Thermal Conductivity (z)	ASTM C714	W/m-K	4.67	4.03	4.03
Thermal Diffusivity	ASTM C714	cm <sup>2</sup> /s	0.039	0.033	0.033
Electrical Resistivity (x,y)	ASTM C611	μΩm	7.8	8	11
Electrical Resistivity (x,y)	GTI Internal	μΩm	NA	10	9
Electrical Resistivity (z)	GTI Internal, 1-Ply	μΩm	NA	934	937
Contact Resistance	GTI Internal	μΩcm²	NA	2.1	3.0
Electrical Conductivity (x,y)	GTI Internal	S/cm	1470	1002	1111
Electrical Conductivity (z)	GTI Internal, 1-Ply	S/cm	NA	10.7	10.7
Thermal Expansion Coefficient (x,y)	<b>ASTM E1545</b>	μm/m-K	1.31	0.95	0.98
Thermal Expansion Coefficient (z)	<b>ASTM E1545</b>	μm/m-K	97.2	81.8	74.1
Flexural Strength, -40 °C	ASTM D790	MPa	63.9	67.3	69.0
Flexural Strength, 23 °C	ASTM D790	MPa	57.5	58.7	61.8
Flexural Strength, 100 °C	ASTM D790	MPa	37.8	47.8	51.3
Flexural Strength, 120 °C	ASTM D790	MPa	NM	44.3	49.7
Tensile Strength, -40°C	ASTM D638	MPa	41.9	41.3	44.6
Tensile Strength, 23°C	ASTM D638	MPa	38.6	37.4	43.8
Tensile Strength, 100°C	ASTM D638	MPa	29.2	32.8	36.4
Tensile Strength, 120°C	ASTM D638	MPa	NM	32.6	37.4
NA - Not Available					
NM - Not Measured					



## **Subtask 3.3: Long-Term Environmental Cycling**

USCAR - III Environmental Test Protocol (Modified)							
	Shoc	k Test	Normal Cycle				
Cycles	100		40				
Step	1	2	1	2	3		
Temperature, °C	125	-40	-40	87.5	125		
Dwell, hrs	0.5		0.5				
Ramp Rate, °C/min	-328	328	4.25	1.25	-2.75		
Hold Temp, °C	-40	125	87.5	125	-40		
Relative Humidity, %	50	NA	80-90	NA	NA		
Dwell, hrs	0.5	0.5	4	1.5	0.5		



## **Subtask 3.3: Long-Term Environmental Testing**

		_	kural dulus	Flex Strer																	
			fore,	Befo	•	Flex			Cycle,	Fle		I Stren	_	F		Modu		Fle		l Stren	_
Resin	Ply	_	psi	ps				lpsi				le, psi				k, Mps			_	ck, psi	
		Avg	Std	Avg	Std	Avg	Std	Delta	t-Test	Avg	Std	Delta	t-Test	Avg	Std	Delta	t-Test	Avg	Std	Delta	t-Test
2G	1	2.88	0.200	8323	462	2.49	0.105	-14%	8.45	8870	229	7%	-5.20	2.22	0.139	-23%	13.4	8775	324	5%	-3.93
2G	3	3.06	0.059	8785	350	2.40	0.158	-22%	19.21	8610	290	-2%	1.89	2.35	0.081	-23%	34.8	8705	253	-1%	0.91
2H	1	3.07	0.196	8793	475	2.47	0.170	-20%	11.37	9138	160	4%	-3.37	2.26	0.174	-26%	15.0	8868	80	1%	
2H	3	3.36	0.112	9063	385	2.36	0.072	-30%	37.01	8770	184	-3%	3.36	2.35	0.180	-30%	23.4	8390	401	-7%	5.92
Paired	t-Te	st (Be	efore-A	After)					5.21				0.53				10.3				0.24
Critica	al t-va	alue							2.447				2.447				2.447				2.447
			nsile dulus	Ten: Strer																	
		Ref	fore		•	Tens	sile Ma	dulus	Cycle	Tensi	le Sti	renath	Cycle	Tens	ile Mo	dulue 9	Shock	Т	neila	Stren	ath
Resin	Ply		fore, psi	Befo	ore,	Tens		dulus Ipsi	Cycle,	Tensi		rength psi	Cycle,	Tens		dulus S Ipsi	Shock,	Te		Stren	_
Resin	Ply		psi	Befo	ore, si			lpsi	Cycle, t-Test			psi	Cycle, t-Test			psi	Shock, t-Test		Sho	ck, psi	_
Resin 2G	Ply 1	M Avg	psi <sup>′</sup> Std	Befo ps Avg	ore, si std	Avg	M	lpsi	t-Test		Std	psi	t-Test	Avg	M	psi			Sho Std	ck, psi	t-Test
	Ply 1 3	<b>Avg</b> 3.91	psi <sup>′</sup> Std	Before ps Avg 4035	ore, si std	Avg	<b>Std</b> 0.844	lpsi Delta	t-Test 0.46	Avg	<b>Std</b> 602	psi Delta	t-Test	Avg	M Std	psi Delta	t-Test	Avg	<b>Sho Std</b> 660	ck, psi Delta	t-Test
2G 2G 2H	1	<b>Avg</b> 3.91	<b>Std</b> 0.573	Befo ps Avg 4035 5638	ore, si std 587 337	<b>Avg</b> 3.81 4.55	<b>Std</b> 0.844	Delta -2% -7%	t-Test 0.46 2.67	<b>Avg</b> 4443	<b>Std</b> 602 412	psi Delta 10%	t-Test -2.38	<b>Avg</b> 3.31 4.67	<b>Std</b> 0.442	psi Delta -15%	t-Test 4.03	<b>Avg</b> 4958 5490	<b>Sho Std</b> 660 200	Delta 23% -3%	t-Test -5.12 1.84
2G 2G	1	Avg 3.91 4.90 5.71	<b>Std</b> 0.573 0.319 0.639	<b>Befo ps Avg</b> 4035 5638 5365	std 587 337 347	<b>Avg</b> 3.81 4.55 3.88	Std 0.844 0.564	Delta -2% -7% -32%	t-Test 0.46 2.67 13.11	<b>Avg</b> 4443 5483	<b>Std</b> 602 412 455	Delta 10% -3% -2%	t-Test -2.38 1.43	<b>Avg</b> 3.31 4.67 3.46	<b>Std</b> 0.442 0.379	<b>Delta</b> -15% -5%	<b>t-Test</b> 4.03 2.35 16.89	<b>Avg</b> 4958 5490	<b>Sho Std</b> 660 200 535	Delta 23% -3% -1%	t-Test -5.12 1.84 0.29
2G 2G 2H 2H	1 3 1 3	Avg 3.91 4.90 5.71 5.21	<b>Std</b> 0.573 0.319 0.639	Avg 4035 5638 5365 6635	std 587 337 347	<b>Avg</b> 3.81 4.55 3.88	Std 0.844 0.564 0.241	Delta -2% -7% -32%	t-Test 0.46 2.67 13.11	<b>Avg</b> 4443 5483 5263	<b>Std</b> 602 412 455	Delta 10% -3% -2%	t-Test -2.38 1.43 0.88	<b>Avg</b> 3.31 4.67 3.46	Std 0.442 0.379 0.127	<b>Delta</b> -15% -5% -39%	<b>t-Test</b> 4.03 2.35 16.89	<b>Avg</b> 4958 5490 5328	<b>Sho Std</b> 660 200 535	Delta 23% -3% -1%	t-Test -5.12 1.84 0.29
2G 2G 2H 2H	1 3 1 3 t-Te	Avg 3.91 4.90 5.71 5.21 est (Be	<b>Std</b> 0.573 0.319 0.639 0.387	Avg 4035 5638 5365 6635	std 587 337 347	<b>Avg</b> 3.81 4.55 3.88	Std 0.844 0.564 0.241	Delta -2% -7% -32%	t-Test 0.46 2.67 13.11 1.69	<b>Avg</b> 4443 5483 5263	<b>Std</b> 602 412 455	Delta 10% -3% -2%	t-Test -2.38 1.43 0.88 8.64	<b>Avg</b> 3.31 4.67 3.46	Std 0.442 0.379 0.127	<b>Delta</b> -15% -5% -39%	t-Test 4.03 2.35 16.89 6.93	<b>Avg</b> 4958 5490 5328	<b>Sho Std</b> 660 200 535	Delta 23% -3% -1%	t-Test -5.12 1.84 0.29 12.62
2G 2G 2H 2H Paired	1 3 1 3 t-Te	Avg 3.91 4.90 5.71 5.21 est (Be	95i Std 0.573 0.319 0.639 0.387 efore-A	Avg 4035 5638 5365 6635	std 587 337 347	<b>Avg</b> 3.81 4.55 3.88	Std 0.844 0.564 0.241	Delta -2% -7% -32%	t-Test 0.46 2.67 13.11 1.69 1.58	<b>Avg</b> 4443 5483 5263	<b>Std</b> 602 412 455	Delta 10% -3% -2%	t-Test -2.38 1.43 0.88 8.64 0.76	<b>Avg</b> 3.31 4.67 3.46	Std 0.442 0.379 0.127	<b>Delta</b> -15% -5% -39%	t-Test 4.03 2.35 16.89 6.93 2.21	<b>Avg</b> 4958 5490 5328	<b>Sho Std</b> 660 200 535	Delta 23% -3% -1%	t-Test -5.12 1.84 0.29 12.62 0.39
2G 2G 2H 2H Paired	1 3 1 3 t-Te	Avg 3.91 4.90 5.71 5.21 est (Bealue	95i Std 0.573 0.319 0.639 0.387 efore-A	Avg 4035 5638 5365 6635 After)	std 587 337 347	<b>Avg</b> 3.81 4.55 3.88	Std 0.844 0.564 0.241	Delta -2% -7% -32%	t-Test 0.46 2.67 13.11 1.69 1.58	<b>Avg</b> 4443 5483 5263	<b>Std</b> 602 412 455	Delta 10% -3% -2%	t-Test -2.38 1.43 0.88 8.64 0.76	<b>Avg</b> 3.31 4.67 3.46	Std 0.442 0.379 0.127	<b>Delta</b> -15% -5% -39%	t-Test 4.03 2.35 16.89 6.93 2.21	<b>Avg</b> 4958 5490 5328	<b>Sho Std</b> 660 200 535	Delta 23% -3% -1%	t-Test -5.12 1.84 0.29 12.62 0.39
2G 2G 2H 2H Paired	1 3 1 3 t-Te	Avg 3.91 4.90 5.71 5.21 est (Bealue	95i 95td 0.573 0.319 0.639 0.387 efore-A	Avg 4035 5638 5365 6635 After)	std 587 337 347	<b>Avg</b> 3.81 4.55 3.88	Std 0.844 0.564 0.241	Delta -2% -7% -32%	t-Test 0.46 2.67 13.11 1.69 1.58	<b>Avg</b> 4443 5483 5263	<b>Std</b> 602 412 455	Delta 10% -3% -2%	t-Test -2.38 1.43 0.88 8.64 0.76	<b>Avg</b> 3.31 4.67 3.46	Std 0.442 0.379 0.127	<b>Delta</b> -15% -5% -39%	t-Test 4.03 2.35 16.89 6.93 2.21	<b>Avg</b> 4958 5490 5328	<b>Sho Std</b> 660 200 535	Delta 23% -3% -1%	t-Test -5.12 1.84 0.29 12.62 0.39



# Accomplishments – Task 4 (Complete)

#### **Machining and Embossment of Small-Scale Composites**

Contributors: GrafTech, Huntsman, Ballard

**Subtask 4.1: Fabricate New Composite Materials** 

**Subtask 4.2: Validate Properties of New Graphite Containing Composites** 

- Mechanical testing results on resin flexible graphite composite samples in 1-, 3-, and 5 ply sheets were obtained.
- Results were equivalent or better then those for the incumbent GRAFCELL standard resin composite system.

**Subtask 4.3: Machined Plates for Single Cell Testing** 

- Machined flow field plate design selected for use in single cell testing.
- Machining of the composite plates for single cell testing is completed.

Subtask 4.4: Design, Fabricate, and Evaluate Small Embossed Test Plates

- Flow field pattern based on a proprietary oxidant flow field die
- Plates were molded to a single plate thickness of less then 0.8 mm.
- Nitrogen gas permeability, in-plane and through plane electrical resistance, and dimensional processing changes (growth factors) have been measured on each plate.
- One of the graphite starting materials was eliminated from consideration based on significantly higher gas permeability results



## **Mechanical Testing t-Test vs. GRAFCELL**

		Flexural N	Modulus,	Flexural	Strength,		
Ply	Temp., °C	Mpsi		p:	si	Flexural Stain, in/in	
		2G	2H	2G	2H	2G	2H
1	-40	-1.00	-0.76	3.11	1.26	-5.67	-12.50
3	-40	7.65	9.26	1.72	8.34	-23.00	-44.00
5	-40	6.03	4.37	0.64	4.58	-6.50	-11.00
1	23	-1.12	0.80	1.36	3.31	-1.50	-2.00
3	23	38.57	24.64	1.51	2.71	-437.52	-951.84
5	23	6.76	78.00	-0.37	2.62	-7.50	-6.33
1	100	18.52	20.62	13.32	9.78	-45.50	-19.80
3	100	12.49	27.64	18.51	9.24	-27.33	-47.00
5	100	13.02	13.58	6.84	37.41	-12.00	-39.00
1	120	17.39	29.76	7.16	22.38	-26.25	-33.00
3	120	14.32	23.54	10.20	7.97	-43.00	-46.00
5	120	17.13	13.56	26.90	40.46	-70.00	-24.00
Criti	cal Value	2.35	2.35	2.35	2.35	2.35	2.35
Pair	ed t-Test (2	2G-2H)					
P-va	alue	0.02		0.00		0.00	
Criti	ical Value	0.05		0.05		0.05	
Cod	е	Improved	Same	Degraded			

Table values are t-Test results for a 95% confidence interval with 3 degrees of freedom



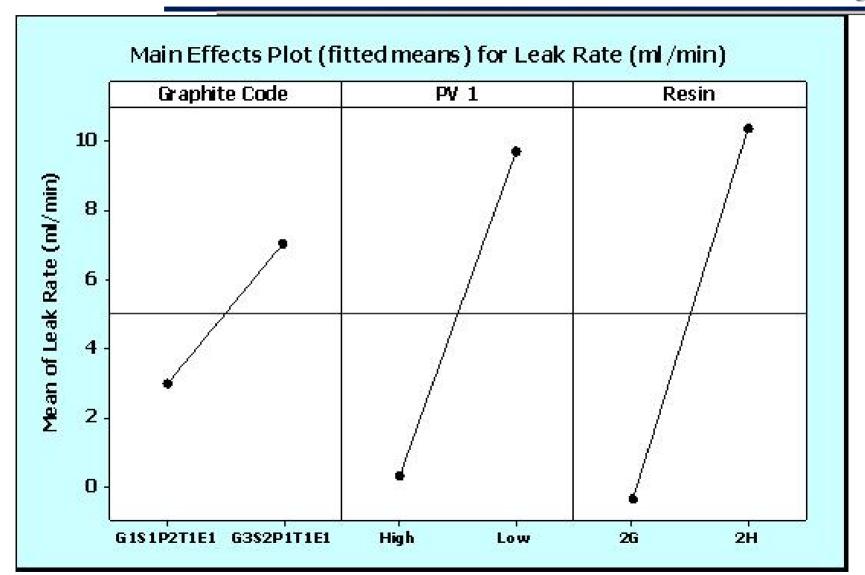
## Mechanical Testing t-Test vs. GRAFCELL

		Tensile N	lodulus.	Tensile S	Strength,		
Ply	Temp., °C	Mpsi		p:	si	Tensile Strain, %	
		2G	2H	2G	2H	2G	2H
1	-40	1.66	4.97	-0.80	4.76	-5.00	-2.73
3	-40	1.35	-1.69	-0.25	5.65	-4.14	-5.50
5	-40	-0.89	-9.14	3.39	0.99	-20.00	-8.75
1	23	2.60	8.03	-2.65	3.11	-6.67	-13.33
3	23	2.69	3.85	1.22	12.51	-274.16	-252.22
5	23	0.12	4.12	3.34	23.68	-3.03	-12.73
1	100	20.63	6.31	2.69	17.45	-11.49	-55.00
3	100	-1.47	-3.47	5.09	7.88	-14.26	-15.48
5	100	-0.23	-2.22	15.33	64.22	-12.75	-18.28
1	120	5.95	5.10	35.14	12.40	-19.74	-12.10
3	120	-20.29	-17.95	7.17	14.50	-16.34	-20.29
5	120	-2.49	-1.83	34.95	109.80	-13.41	-12.79
Critica	al value	2.353	2.353	2.353	2.353	2.353	2.353
Paired	l t-Test (2G	-2H)					
P-valu	ie	0.21		0.00		0.92	
Critica	al Value	0.05		0.05		0.05	
Code		Improved	Same	Degraded			

Table values are t-Test results for a 95% confidence interval with 3 degrees of freedom

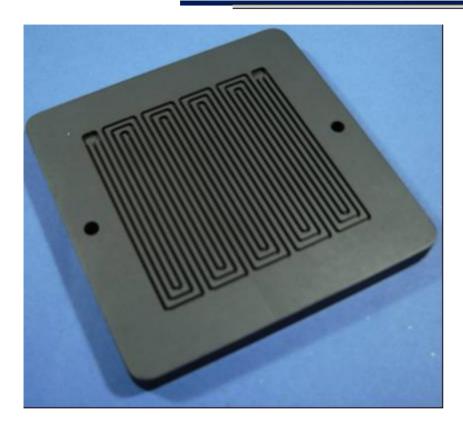


#### **Subtask 4.4: ANOVA Results on Gas Permeability**





#### **Benzoxazine Resin GRAFCELL Composite Plates**



Machined GRAFCELL Composite Single Cell Flow Field Plate



Molded GRAFCELL Composite Corrugated Flow Field Oxidant Plate



# Accomplishments – Task 5 (In Progress)

#### Single Cell Testing

Contributors: GrafTech, CWRU

Subtask 5.1: Select Fuel Cell Components Suitable for High Temperature Testing (Compete)

- High temperature cell components identified and procured
- Difficulty in obtaining high-temperature MEA delayed the start of single cell testing

Subtask 5.2: Develop Test Method for Analysis of Fuel Cell Leachate (In Progress)

Test methods defined and analysis is in progress

Subtasks 5.3 - 5.5: Set up and Conduct 1000-hr Single Cell Test (In Progress)

- Single cell testing has begun
- 2G resin composite plate has operated for 300 hrs as of 4/11/08 Subtask 5.6: Post Test Plate Analysis (Not started)



## Single Cell Testing

#### **Protocol**

- ETEK 1500 GDL
- 114 hrs @ 80 °C (70% RH)
- 86 hrs @ 120 °C (24% RH)
- Cells conditioned 24-48 hrs @ 80 °C
- Liquid samples collected.
- Cell Resistance:
  - After 114 hrs @ 80 °C:
     0.23 Ohm cm²
  - After 71 hrs @ 120 °C:
     0.55 Ohm cm²
  - After 86 hrs @ 120 °C:
     0.54 Ohm cm²

#### Results

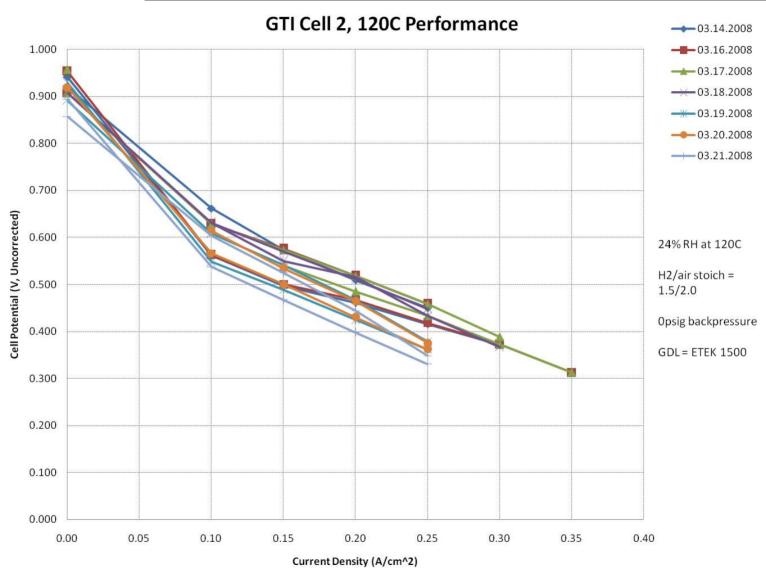
- 2G plate: 352 hours operation at 120°C
- Typical MEA lasts approximately 100 hours.

#### **Cell Operation Times**

- Cell 1: 98.5 hours
- Cell 2: 117 hours
- Cell 4: 136.5 hours



#### **Fuel Cell Performance High Temperature MEA**





# Accomplishments – Task 6 (In Progress)

#### **Design and Manufacture Full-size Bipolar Plates**

Contributors; GrafTech, Ballard

**Subtask 6.1: Develop Test Methods and Test Plate Coolant Durability (Complete)** 

- Ballard has developed a glycol permeation test based on ASTM D739-99a to evaluate the permeation of ethylene glycol across the resin expanded graphite composite.
- Permeation testing has been completed on the 2G and 2H composites and the results are being reviewed. The data has been submitted to GrafTech for consideration to the overall material down-selection.

**Subtask 6.2: Review of Existing Flow Field Plate Architectures (Complete)** 

 Ballard has reviewed the DoE requirements for this project task and evaluated existing Ballard fuel cell architectures as well as GrafTech material properties to arrive at a proposed design.

**Subtask 6.3: Design Flow Field Plate Using Existing Architectures (in progress)** 

- Ballard has submitted to GrafTech a proposed fuel cell plate design in the form of electronic drawings and solid models files. The proposed design has incorporated known design features to aid in part formation during processing.
- The proposed design has a plate assembly thickness below 1.6mm and a plate active area greater than 250cm<sup>2</sup>.

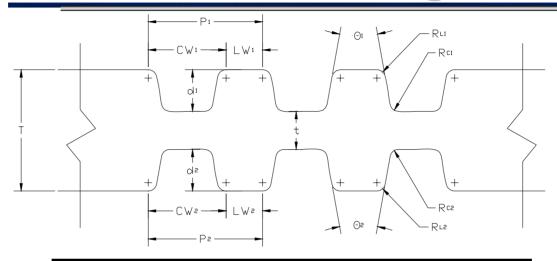
**Subtask 6.4: Fabricate Full Size Embossing Die Set (Not Started)** 

**Subtask 6.5 Emboss Full-size Bipolar Plates (In progress)** 

- Existing Press for use in full-size bipolar plate fabrication were evaluated and final selection has been made
- Final review and selection of graphite, resin and processing conditions has been conducted. 2G resin system and G3 graphite mat selected
- Manufacture of resin for full size plate production has been scheduled



# **Typical Flow Field Plate Design Parameters**



Parameter Definition	Parameter
Web Thickness	t
Channel Depth	$d_n$
Landing Width	LW <sub>n</sub>
Channel Width	CW <sub>n</sub>
Pitch	$P_n$
Draft Angle	$\Theta_{n}$
Landing Crown Radius	$RL_n$
Channel Root Radius	RC <sub>n</sub>
Overall Thickness	Т



## Future Work – 2008 and early 2009

Task 6: Design Flow Field Plate Using Existing Architectures (Q2 & Q3 2008)

- Finalize plate design after reviews including minimum web thickness and volume uniformity
- Build small test tools to calculate growth factors and assess formability
- Final plate design review with material selection and growth factors incorporated
- Build full size embossing tool and initiate material fabrication
- Build leak check device and glue fixtures
- Commission glue equipment
- Build compression stack hardware including all supporting components
- Select Membrane Electrode Assembly (MEA) and modify seal equipment
- Fabricate, inspect and glue plates assemblies

Task 7: Short Stack Test of Full-size Plates (Q4 2008 & Q1 2009)

- Fabricate and seal MEAs
- Assemble inspected and glued plates with sealed MEAs in compression stack hardware
- Commission test station with duty cycle
- Conduct durability testing targeting 1000 hours on a 10 Cell stack
- Conduct freeze start testing
- Post test analysis, results and review including plate inspection
- Deliver Full Size Plate Stack to DOE

Task 8: Economic Assessment of New Technologies

Q3 & Q4 2008

**Task 9: Final Report Preparation** 



# **Summary**

- All critical starting material evaluation and testing is complete
- Graphite mat and resin system for full size plate fabrication have been selected.
- New composite systems have been shown to have equivalent or improved dimensional stability and mechanical and thermal properties over the current GRAFCELL composite.
- Gas impermeability has been demonstrated to a single plate thickness of less then 0.8mm.
- Critical processing parameters for plate embossing have been identified and optimized.
- Basic plate architecture has been identified.
- Production press for fabrication of full-size plates has been identified, evaluated, and certified for use.
- Preliminary leachate, glycol and single cell testing results are positive or do not indicate any significant problems with cell operations at elevated temperature.
- Program is on schedule to produce full-size flow field plates for high temperature short stack testing by Ballard in early 2009

